

Research Article

The Effect of Deposition Rate on Morphology and Structural Properties of Carbon-Nickel Composite Films

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Carbon-nickel films were grown by radio frequency magnetron cosputtering on glass substrates. The films were deposited under different deposition times, from 50 to 600 sec, at room temperature. We noticed that up to 180 sec the sputtering occurs in more metal content mode and in greater than 180 sec it occurs in more nonmetal content mode. It is shown that the structural and morphological properties of carbon-nickel films were strongly influenced by this behavior.

1. Introduction

Recently, a lot of interest has been given to amorphous carbon (a-c) metal-containing films a-c: Me (Me = Au, Ag, Cu, Mn, ...) due to their low cost and wide variety of properties [1, 2]. Previous studies on metal-containing carbon films have been mainly focused on hydrogenated a-c films which are produced by physical vapor and chemical vapor deposition techniques (PVD and CVD, resp.) involving either magnetron sputtering or cathodic arc deposition of metal phase in a reactive hydrocarbon gas environment [3]. However, less attention has been paid to the nonhydrogenated a-c films. Usually hydrogen-free a-c: Me films have been prepared by PVD techniques such as RF-magnetron sputtering in which a multicomponent metal-graphite target is used [4, 5]. An element such as Ni with low or without affinity to carbon atom leads to a relatively sharp interface between the carbon matrix and the metallic phase and therefore distinct properties are produced in comparison to carbide forming elements [6, 7]. Moreover, carbon-nickel composite up to now are studied for their interesting properties such as residual stress reduction for hard films [8], decrease of the friction coefficient [8], or improvement of the dielectric constant [9]. In the previous reports, the dependence of these properties on the deposition times, nickel content, the substrate temperatures, and deposition parameters mainly at constant deposition rate was investigated. However, it has been paid less attention to the effect of different deposition rates on films deposited using RF-magnetron sputtering. In the present work, we investigated the effect of deposition times with different deposition rates on structure and morphology properties of amorphous C-Ni composite films in some more detail.

2. Experimental Details

Carbon-nickel composite films have been prepared by RFmagnetron co-sputtering on glass substrates using a multicomponent target (10 cm in diameter) consisting of pure graphite (99.99 at %) and strips of pure nickel approximately 2 cm^2 attached to the graphite race track, that corresponds to approximately 2.5% in area. Before loading in the deposition chamber, the substrates were ultrasonically cleaned in acetone bath for 20 min and then were dried in hot air flow. The films were grown at room temperature in a deposition chamber evacuated to a base pressure 5 mbar, and then the constant Ar working pressure of mbar was settled



FIGURE 1: XRD spectra of the C-Ni films as a function of deposition times.

and maintained by throttle valve. Deposition was done in constant RF power regime 400 w. The angle of incident ions with target surface was 90°, and the substrate-target distance was 60 mm. We prepared films at different deposition times 50, 90, 180, and 600 sec and at different deposition rates 0.84, 1.6, 1.71, and 1 nm/sec, respectively. The thickness of the films was measured by Tencor Alpha-Step 500 profiler. AFM analysis on noncontact mode was used to obtain surface morphology properties. The average nanoparticle size and root mean square (RMS) roughness were obtained from AFM data (WSXMsoftware-2007). The crystal phase was determined using X-ray diffractometry (XRD) with Cu K α radiation. The RBS spectra were obtained using incident ions (4He) with energy of 2 keV. The atomic content of the films was obtained from RBS data by SIMNRA software simulation.

3. Results and Discussion

Figure 1 shows XRD patterns of the co-sputtered C-Ni films at different deposition times. As shown in Figure 1, it is found that carbon films exhibit an amorphous structure while metallic nanoparticles are either amorphous or crystalline. With increasing deposition time from 50 to 180 sec, the nickel clusters show amorphous behaviors while, from 180 to 600 sec, nickel clusters have nearly crystalline structures. Such behavior for carbon films embedded by nanosized metal particles was reported by Wu and Ting [10]. As the time of deposition increases the feature of amorphous structure diminishes, while crystalline peaks of metal appear. It is therefore believed that a sufficient time and amount for metallic constituent are required to allow the metal to form crystalline structure. In the low deposition rate and high deposition time, Ni atoms have enough time to diffuse and improve the crystallinity.

Figure 2 shows AFM images of the films that are recorded over scan area. The surface topography is traditionally analyzed by surface roughness measurements such as rootmean-square (RMS) roughness which can be expressed as

TABLE 1: Details of AFM analysis of C-Ni composite films.

Deposition time (sec)	Thickness (nm)	Mean height (nm)	RMS (nm)	Deposition rate (nm/sec)
50	42	9	3.5	0.84
90	150	13	4.9	1.6
180	309	20	5	1.71
600	608	16	4	1

 $R_{\rm rms} = \left((1/N)\sum_{i=1}^{N} |Z_i - \overline{Z}|^2\right)^{1/2}$, where Z_i is the measured height of *i*th point in the scan area, \overline{Z} is the average of height, and N is the number of all points defined in the scanned area [11, 12]. The microstructure and topographical details of a thin film depend on the kinetics of growth and hence on the substrate temperature, the source and energy of impurity species, the chemical nature, the topography of the substrate, and ambient gas [13]. With increasing deposition time and hence the thickness, the mean height is also expected to increase. The mean height of the films deposited at 50, 90, 180, and 600 sec estimated to be 9, 13, 20, and 16 nm, respectively, and their deposition rates are calculated (by dividing the film thickness over deposition time), are found to be 0.84, 1.6, 1.71, and 1 nm/s, respectively. The deposition rate in more metallic content mode is greater than that of the nonmetallic content mode [13]. In the low deposition rate and high deposition time, there are some adsorptions atoms which have enough time to migrate to sites where the surface energy is low enough to be covered by the coming atom and as a result the film surface is very smooth [12]. As shown in Table 1 the RMS roughness of the films deposited at 50, 90, 180, and 600 sec is 3.5, 4.9, 5, and 4 nm, respectively. With increasing deposition time from 50 to 180 sec, the deposition rate is increased and from 180 to 600 sec, the deposition rate is decreased. Therefore, up to 180 sec the sputtering occurs in more metal content mode and in greater than 180 sec the sputtering occurs in more nonmetal content mode, and thereby we expect that up to 180 sec the RMS roughness is increased and in greater than 180 sec the RMS roughness is decreased. It is expected that the mean height of the film deposited at 600 sec is either greater than or, at least, equal to the mean height of the film deposited at 180 sec. In spite of the fact that the thickness of the film deposited at 600 sec is greater than that of the film deposited at 180 sec, it is shown that the mean height of the film deposited at 600 sec is less than the mean height of the film deposited at 180 sec. Therefore, the effect of deposition rate on the mean height is greater than the effect of deposition time.

Figure 3 shows RBS spectra (experiment and simulation results) of nickel-carbon composite films deposited at 600 sec. The steps at about 400, 500, and 750 keV are due to C, O, and Si nuclei, respectively. The peak between 1000 and 1500 kev is attributed to Ni nuclei [14]. The results of SIMNRA software simulation of deposited film showed that the layer with thickness 3300 (1E15atom/cm²) contains 31% Ni.



FIGURE 2: Three-dimensional AFM images of C-Ni films deposited at (a) 50, (b) 90, (c) 180, and (d) 600 sec.



FIGURE 3: RBS spectra, experimental and simulated results of C-Ni film deposited at 600 sec.

4. Conclusions

Nanocomposite thin films of nickel nanoparticles embedded in amorphous carbon matrix were fabricated using RF-magnetron co-sputtering from a multicomponent target (10 cm in diameter) consisting of pure graphite (99.99%) and strips of pure nickel approximately 2 cm² attached to the graphite race track. It has been observed that the deposition rate plays an important role on the structural and morphological properties of C-Ni composite films. It is shown that the prepared films at deposition rate 1.71 nm/s have the maximum mean height of 20 nm and the maximum roughness of 5 nm. It is also shown that the crystallinity of the films depended on the deposition rate and deposition time.

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